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José L. Mendoza, jmendoza@chem.ucla.edu[1], Hani M. El-Kaderi, hkaderi@chem.ucla.edu[2], Joseph R Hunt, huntjose@chem.ucla.edu[3], Adrien P Côté, apcote@chem.ucla.edu[2], and O. M. Yaghi, yaghi@chem.ucla.edu[4]. (1) Department of Chemistry and Biochemistry, University of California, Los Angeles, 607 Charles E. Young Drive East, Los Angeles, CA 90095, (2) Department of Chemistry and Biochemistry, Center for Reticular Chemistry at the California NanoSystems Institute, University of California Los Angeles, 607 Charles E. Young Drive, East, Los Angeles, CA 90095-1569, (3) Department of Chemistry and Biochemistry, Center for Reticular Chemistry at the California NanoSystems Institute, University of California, Los Angeles, 607 Charles E. Young Drive East, Los Angeles, CA 90095, (4) Department of Chemistry and Biochemistry, University of California-Los Angeles, 607 Charles E. Young Drive, East, Los Angeles, CA 90095

The first members of covalent organic frameworks (COF) have been designed and successfully synthesized by condensation reactions of phenyl diboronic acid C6H4[B(OH)2]2 and hexahydroxytriphenylene C18H6(OH)6. The high crystallinity of the products (C3H2BO)6 (C9H12)1 (COF-1) and C9H4BO2 (COF-5) has allowed definitive resolution of their structure by powder X-ray diffraction methods which reveal expanded porous graphitic layers that are either staggered (COF-1, P63/mmc) or eclipsed (COF-5, P6/mmm). They exhibit high thermal stability (to temperatures up to 500- to 600-C), permanent porosity, and high surface areas (711 and 1590 m2/g, respectively) surpassing those of related inorganic frameworks. A similar approach has been used for the design of other extended structures.